GREEN SYNTHESIS OF DUAL DOPED ZINC OXIDE (ZnO) NANOPARTICLES VIA MICROWAVE IRRADIATION FOR MULTIFUNCTIONAL APPLICATIONS

SYNOPSIS

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Submitted by

NirdoshVerma
Enrollment No.: H220898

Under the supervision of

Co-Supervisor

Dr. Dinesh Pathak
Department of Physics,
The University of the West Indies,
St Augustine Campus, Trinidad.

Supervisor

Dr. Naveen Thakur
Professor
Department of Physics.
Career Point University, Hamirpur.

DEPARTMENT OF PHYSICS
SCHOOL OF BASIC & APPLIED SCIENCES
CAREER POINT UNIVERSITY HAMIRPUR
TIKKER (KHARWARIAN), BHORANJ
HAMIRPUR (HP)-176041, INDIA

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1. Introduction

1.1 Nanotechnology

Nanotechnology is widely spreading in all areas of technology that are strongly linked with today's lifestyle. Richard Feynman, a Nobel Prize winner, defined the concept of nanotechnology in 1959. He states that "there's Plenty of Room at the Bottom" at the annual meeting of the American Physical Society at Caltech. In his hypothesis, he said that it couldn't be possible to write all the information of Encyclopedia Britannica on the top of a pin and defined the idea of making smaller machines with the help of machines at the atomic or molecular level (Feynman et al., 2011). In 1974, the term nanotechnology was used by Professor Norio Taniguchi, who defined it as "processing, separation, consolidation, and deformation" of bulk material through atoms or molecules. Nanotechnology is today collaborating with all fields of science for the impressive utility of its application for the welfare of mankind. Moreover, its smaller size and high surface-to-volume ratio make it possible to utilize it in sensors, rubbers, protection from UV rays, coatings, storage devices, diagnosis and cancer treatments, and other applications for enhancing the strengths and capabilities of materials. It also provides solutions to various environmental and technological problems in solar cells, water treatment, air pollution, medicine, etc.

1.2 Nanoparticles

These are matter's particles that have sizes ranging from 1 to 100 nm and are usually called nanoparticles or ultrafine particles (Hartland et al., 2013). Particle sizes smaller than 1 nm are considered atoms or clusters. The concept of nanoparticles does not originate from the modern era, but it has evidence of its origin in ancient times. Ancient China utilized the nanoparticle for ink production. Official nanoparticles originated when modern science actually reached the nanoscale. Furthermore, these nanoparticles have industrial as well as medical uses like nanocoating materials to design RAM (Radio Absorbent Materials) and to diagnose and treatment of various diseases. Metal oxide nanoparticles (e.g., TiO_2, ZnO) exhibit unique properties for advanced applications in biomedicine, renewable energy, environmental remediation, and nanoelectronics, enabling breakthroughs in drug delivery, solar cells, pollution control, and miniaturized devices.

1.3 Zinc Oxide (ZnO)

ZnO has high photo stability, high chemical stability, and a higher electrochemical coupling coefficient than other metal oxides, which makes it more advantageous for a number of applications (Parihar et al., 2018; Dhayagude et al., 2017). Zinc oxide is used in electronic devices, lasers, sensors, and other technologies because it has high thermal and mechanical stability with a wide band gap energy of 3.37 eV and a high bond energy of 60 meV (Bilgiliet al., 2019; Kalpana et al., 2017). Due to its unique properties, researchers are showing interest in ZnO for various nanostructured applications. Various sizes and shapes of ZnO nanoparticles have already been synthesised, like springs and rings (Lalithamba et al., 2018), needles (Gunathilaka et al., 2021), nanorods (Mintcheva et al., 2018; Mohan et al., 2020; Mohamed et al., 2019), nanoplates (Khatami et al., 2018; Im et al., 2016), wires (Nikoobakht et al., 2013; Moradpoor et al., 2013; A Barzinjy et al., 2020), snowflakes (Sirwlkhatim et al., 2015), etc. Biosynthesized ZnO nanoparticles produced using plant extracts demonstrate remarkable biological and catalytic properties. These eco-friendly nanomaterials show enhanced performance in medical, environmental, and agricultural applications due to their unique surface chemistry and improved biocompatibility compared to conventional synthesis methods. Table 1 represents the synthesis methods, conditions, and morphologies of the ZnO nanoparticles along with the usage of plant extract.

Table 1: Synthesis of zinc oxide nanoparticles by using different techniques

S. No	Techniques	Chemicals used/plant	Fabrication Conditions	Size and shapes	Ref
01	Solvothermal method	Zinc acetate, Zinc nitrate, ethanol, imidazoliumtetrafluorobarate ionic liquid	Reaction temperature (150-180^{0}C), drying at 80^{0}C, calcination at 500^{0}C.	Hollow micro sphere (2-5 micro meter), flower (average size 2.5 x 10^{-6} m),nano rods (average size 20nm),	(Zhang et al, 2010)
02	Sol-gel method	Zinc acetate, ethanol, oxalic acid and methanol	Reaction temperature (60^{0}C), calcination at 500^{0}C, drying at 80^{0}C for 24h,	Rod shape (diameter 100 nm with average length 150-200 nm)	(Nikoobakht et al, 2013)

03	Mechanochemical process	$ZnCl_2$, NaCl Na_2CO_3.	Calcination at 600^0C for 2 h	Hexagonal structure with diameter (21 -25 nm)	(Kong et al, 2004)
04	Precipitation process	Zinc Nitrate, NaOH	Synthesis for 2h, drying at 100^0C for 1h ,reaction time 2h	Spherical (size 400nm)	(Wu et al, 2002)
05	Green synthesis (co-precipitation)	*Azadirachta indica* (Neem) extract	60°C, pH 11, 2 hr reaction	Spherical, ~20–30 nm (SEM/XRD)	(Krejcir et al., 2020)
06	Sol-gel	*Aloe barbadensis miller* extract	80°C, 3 hr aging	Hexagonal wurtzite, ~50 nm (TEM)	

1.4 *Psidium guajava*

Psidium guajava belongs to the myrtle family and is commonly known as guava (Biswas et al., 2013). Traditionally, it has been used as a medicine ingredient to heal wounds. Although guava is also considered a medical plant because it shows properties like anti-bacterial, anti-oxidant, anti-diabetic, and anti-cough due to its biological function on a large scale (Rehan et al., 2019), Photochemical biological composites such as flavonoids, vitamins, and phenolic compounds (Barbalho et al., 2012) are accompanied by guava leaf extracts for the synthesis of nanoparticles, which act as stabilizing as well as reducing agents (Matinise et al., 2018). Due to its high level of antioxidants, lycopene present in guava leaves decreases the risk of prostate, breast, and oral cancer (Kafle et al., 2018). Lycopene has shown a significant role in declining cancer risk, as reported in various studies. Malaria, diarrhoea, vomiting, ulcers, dysentery, etc. are diseases controlled by several parts of the plant (Awuchi et al., 2020; Ahmed et al., 2021). Synthesis of nanoparticles through the green method related to medical plants results in good anti-fungal, anti-bacterial, anti-oxidant, and other properties. Plant extract is used as a reducing and capping agent in synthesis. Along with it, it was also used to constrain the size and shape of nanoparticles (Khalil et al., 2018).

1.5*Colocasia esculenta*

Colocasia esculenta is commonly known as Taro and has been easily available in many countries for several centuries. It is mainly available in Asia, tropical and sub-

tropical regions, New Zealand, South Africa, and many other countries. *Colocasia esculenta* plants are utilized in various aspects, such as medicines, seasoning, dyes, cosmetics, etc., in order to increase human health and human life. Ethnopharmacological studies on medicinally imported plants attracted investors from all over the world (Arulmozhi et al., 2007). Waste *Colocasiaesculenta* leaf extract was used to prepare copper oxide nanoparticles by using green biosynthesis as their recyclable catalyst (Barman et al., 2021).

1.6*Murraya koenigii*

Murraya koenigii, also called curry leaf, is a small evergreen tree native to India and Sri Lanka, frequently used in South Asian cooking (Datta et al., 2023). Belonging to the Rutaceae family, the plant is famous for its fragrant, dark green, pinnate leaves. These leaves are packed with vital nutrients, such as vitamins, minerals, and antioxidants (Chaudhary et al., 2023). Curry leaves are packed with antioxidants, vitamins (A, B, C), and minerals (iron, calcium, phosphorus), and have long been used in Ayurvedic medicine. They are thought to aid digestion, help regulate blood sugar levels, enhance liver function, and encourage hair growth.

2. Literature Review

Zinc oxide nanoparticle synthesis by *Psidium guajava* leaf extract for antibacterial activity against some bacteria. A microwave-assisted technique was utilized to synthesize nanoparticles via the sol-gel method (Ramya et al., 2022). Characterization techniques such as XRD, SEM, EDAX, and FTIR were performed and analyzed to show that ZnO is hexagonal in phase with a particle size of approx. 15.8 nm. Synthesized nanoparticles in this paper result in anti-bacterial activity for both types of bacteria. Synthesis methods such as wet-chemical, sonication and hydrothermal methods were used to synthesis ZnO nanoparticles with photo constituents of *Psidium guajava* leaf extract (Saha et al, 2018). Physico-chemical, photocatalytic and biological property has been performed and results are compared to get broader understanding about various parameters of ZnO nanoparticles. In this study, nanoparticle size is different for all synthesis techniques. Furthermore, antimicrobial and antibacterial properties were studied to utilize nanoparticle for bio-medical applications. There are some other studies for green synthesis, like the ecofriendly synthesis of ZnO nanoparticles with *Phyllanthus emblica* seed powder (Loganathan et al., 2018). In this study, X-ray diffraction, SEM, TEM, EDAX, and photoluminescence studies (PL) were performed to get an accurate

result for nanoparticles. Microwave-assisted chemical precipitation methods were used to synthesize cysteine-capped ZnO (CCZ) nanospheres (Selvin et al., 2016). In this paper, experimental results confirmed that 50 mg of CCZ photocatalyst produced higher photocatalytic activity than bare ZnO in the removal of RhB dye. Also, particle size reduced with the addition of CCZ, and hence the surface-to-area ratio increased.

Zinc oxide nanoflowers were biologically prepared at low temperatures by using the green method with a cell-free extract of Chlamydomonasreinhardtii (Rao et al., 2016). The nanorods measured 330 nm in length and accumulated together to form a flower structure. Nanoflowers were composed of zinc and oxygen, as confirmed with EDX analysis. Further, algal molecules act as reducing or capping agents for the synthesis of flower-shaped nanostructures. *Atalantiamonophylla* leaf extract was used as a reducing agent to synthesise silver/zinc oxide nanocomposites under the microwave technique (Verma et al., 2021). In this study, the average crystallite size was calculated with the help of XRD results and observed to be 17.37 nm; also, TEM results produced a size of 16.76 ± 10.15 nm. Other results of synthesized nanoparticles encourage their utilization in biomedical, optoelectronic, and other applications.

A microwave-assisted technique was used to synthesize ZnO and Ag-doped ZnO nanoparticles with *Colocasiaesculenta* leaf extract (Kumar et al., 2019). In this study, doped nanoparticles showed antimicrobial properties in a broader area as compared to undoped nanoparticles against bacteria and fungi. Further, both nanoparticles showed zones of inhibition for *E. coli* and *A. oryzae* microbes. Zinc oxide nanoparticles were synthesized with *Aeglemarmelos* as fuel by using the microwave irradiation method (Mallikarjunaswamy et al., 2020). In this paper, methylene blue degradation by using Zno as a photocatalyst was reported to be 95% in 40 min. For antibacterial activity, it was reported that by increasing the concentration of ZnO, antibacterial activity also increases. The synthesis of zinc oxide nanoparticles (ZnO NPs) from the aqueous leaf extract of *Murrayakoenigii* (MK) has been the focus of recent studies (Sharma et al., 2023). These MK ZnO NPs were tested for their in vitro antidiabetic, antioxidant, antimicrobial, and cytotoxic activities. The results showed significant antidiabetic and cytotoxic effects, in addition to moderate antibacterial activity and free-radical scavenging potential.

Ag/ZnOnanocomposites (NCs) were synthesized using eco-friendly, low-cost plant extracts from *Murrayakoenigii* and *Zingiberofficinale*, with additional synthesis of Ag and ZnO nanoparticles (NPs) via chemical methods for comparison (Selvan et al.,

2021). The Ag/ZnO NCs synthesized using *Zingiberofficinale* extract exhibited the highest cytotoxicity against all tested cell lines. Morphological analysis revealed significant apoptotic cell death, further corroborated by elevated levels of reactive oxygen species (ROS) generation. Al–Fe co-doped ZnO (Al–Fe/ZnO) nanorods were fabricated through a straightforward and cost-effective hydrothermal method(Khalid et al., 2019). UV–visible spectroscopy analysis showed that doping with Al and Fe decreased the band gap energy from 3.37 eV to 2.62 eV. The Al–Fe/ZnOphotocatalyst exhibited a degradation rate constant (k) for methylene blue (MB) dye that was 17 times greater than that of pure ZnO.

3. Research Gap Identification

In fact, many researchers have studied the green synthesis of ZnO and doped ZnO nanoparticles by using different elements, but there is very little literature on the co-doped green synthesis of zinc oxide nanoparticles. Further, there is less literature on the utilization of transition and lanthanides metals as dopants on ZnO structure. This work will give the comparative physical and chemical properties of green-synthesized dual-doped ZnO NPs. Along with it; synthesized nanoparticles will be used for antibacterial and antioxidant activity.

4. Objectives

The objectives of the present study based on the above discussion are as follows:

- To study the green synthesis of dual-doped ZnO nanoparticles with the combination of lanthanide and transition metal as dopants by using the microwave technique.
- To characterize the synthesized dual-doped ZnO nanoparticles by using different characterization techniques like X-ray diffraction (XRD), UV-visible spectroscopy, Fourier transform infrared spectroscopy (FTIR), transmission electron microscope (TEM), and scanning electron microscope (SEM).
- To evaluate the antioxidant and antibacterial activities of nanoparticles for their potential applications in wastewater purification, bacterial controls, and other environmental remediation processes.

5. Proposed Methodology

5.1 Materials

Zinc acetate dihydrate, cobalt acetate tetrahydrate, copper acetate monohydrate, gadolinium chloride hexahydrate, cerous chloride heptahydrate, Iron chloride hexahydrate, Aluminum chloride anhydrous powder, sodium hydroxide, and ethanol will be used as chemicals in synthesis. Plant leaves of *Psidium guajava*, *Murraya koenigii* and *Colocasia esculenta* will be collected from local areas of district Hamirpur, Himachal Pradesh.

5.2 Synthesis of plant extract

Plant leaves will be cut into smaller pieces and properly washed under tap water. Plant extract will be prepared by heating 10 g of crushed leaves with 100 ml of DI water at 800 °C for 1 hour. After cooling the resulting solution, it will be filtered to obtain a clear solution. Then, the plant extract will be stored in a refrigerator at 4°C for further experimentation. The same procedure has been followed for both leaf plants.

5.3 Synthesis of co-doped ZnO nanoparticles

Microwave technique will be used for the synthesis of co-doped ZnO nanoparticles. Zinc acetate dihydratewill be dissolve in 100 ml of DI water. Solution will be stir for 60 min using a magnetic stirrer. Then, metal ion as dopant will add to the solution, which will stir for 60 min. Similarprocedure, will be follow for other dopant metal ion. Plant extract as stabilizer will add drop-wise into the solution, which will again stir for 60 min. Solution will be place inside a domestic microwave for 10 min at 200^0C. Precipitates will bepreparedand they will filter out of the solution after cooling. The resultant precipitateswill wash with DI water and ethanol. Then, precipitates will be drying in an oven at 80^0C to obtain co-doped ZnO nanoparticles. Obtain nanoparticles will be crushed to get fine powder for further characterization.

5.4 Characterization of synthesized dual-doped ZnO nanoparticles

The green synthesized dual-doped ZnO nanoparticles will be characterized by XRD to determine their crystallite size, crystal structure, lattice parameters, etc. The bandgap of synthesized nanoparticles will be measured with UV-Vis spectroscopy, and functional groups present inside the nanoparticles will be identified with Fourier transform infrared spectroscopy (FTIR). Surface morphology and particle size will be determined with SEM, EDAX, and TEM analysis.

5.5 Antibacterial activity

Antibacterial activity of dual-doped ZnO nanoparticles will be tested by using the well diffusion method against different positive and negative gram microbes like Escherichia coli (MTCC 739), Staphylococcus aureus (MTCC 737), and Bacillus subtilis

(MTCC 441). Bacterial strains will be purchased from the Institute of Microbial Technology (IMTECH), Microbial Type Culture Collection Centre, Chandigarh.

5.6 Antioxidant activity

The radical scavenging activity of dual-doped ZnO nanoparticles was assessed using the DPPH assay (Kumar et al., 2023). Various concentrations of the synthesized catalyst were combined with a 0.1 mM DPPH solution in methanol. Upon the addition of nanoparticles, the violet color of the DPPH radical solution faded to colorless. After a specified incubation period, absorbance at 517 nm was recorded, with ascorbic acid serving as the standard. A reduction in absorbance reflected an enhancement in DPPH radical-scavenging activity. The efficacy of scavenging DPPH free radicals was determined using the following equation.

$$\% \text{ inhibition} = \frac{(A_0 - A_1)}{A_0} \times 100$$

Absorbance of the solution denoted by A_1, while absorbance of the control solution denoted by A_0.

6. Thesis Outline

The thesis will be comprised of following chapters:

Chapter 1 (Introduction): It will constitute of the brief account about nanotechnology along with characteristics of ZnO nanoparticles and plant extract. It will also include the objectives of proposed study.

Chapter 2 (Literature Review): This chapter will deal with intense literature with context to the synthesis, characterization and applications of ZnO nanoparticles. It will also include the research gap identification.

Chapter 3 (Materials and Methods): This chapter will comprise the detail of materials, chemical as well as green synthesis, characterization techniques and applications of co-doped ZnO nanoparticles.

Chapter 4 (Results and Discussion): This chapter will include the reports regarding physical and chemical properties of wurtzite phase of dual-doped ZnO nanoparticles along with analysis and comparison of the findings the work.

Chapter 5 (Summary and Conclusion): It will include the overall summary and conclusion of the entire study with regard to the work with context to the objectives cited.

Chapter 6 (References): This will include references which will be strictly according to the Harvard style.

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CERTIFICATE
(SYNOPSIS APPROVAL CERTIFICATE)

This is to certify that the Synopsis/Revised Synopsis for Ph.D. Thesis titled, "**GREEN SYNTHESIS OF DUAL DOPED ZINC OXIDE (ZnO) NANOPARTICLES VIA MICROWAVE IRRADIATION FOR MULTIFUNCTIONAL APPLICATIONS**", submitted by Mr. Nirdosh Verma to the Career Point University, Hamirpur (H.P.), India in partial fulfillment of the requirements for the degree of DOCTOR OF PHILOSOPHY in the Division/Department of Physics, School of Basic and Applied Sciences has been approved by the Research Advisory Committee (RAC) after an Oral Examination of the student on dated **09.06.2023**.

Dr. Naveen Thakur
Professor
(Supervisor)

Research Advisory Committee

28/8/23
Dr. Indu Sharma
Associate Professor
(Member)

28/8/23
Dr. Anjali Sharma
Assistant Professor
(Member)

Dr. Naveen Thakur
Professor
(Head of Department)
Department of Physics

Dr. Kuldeep Kumar
Associate professor
(Dean/Assoc Dean R & D)